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Hazardous Air Pollutant Free Replacement for Specification A-A-1936A Contact Adhesive

by Faye R. Toulan, Daniel Stewart, and John J. La Scala

ARL-TR-6831 **February 2014**

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14. ABSTRACT
The goal of the Sustainable Painting Operations for the Total Army (SPOTA) program is to significantly reduce the amount of hazardous air pollutant (HAP) emissions produced in coating operations, including adhesives and sealant application and removal. This work focuses on determining a suitable HAP-free or low-HAP contact adhesive replacement for the A-A-1936A specification. A-A-1936A is a common contact adhesive specification used for commercial and military applications. The current material has a HAP content of 22 weight-percent (wt.%) and 422 g/L volatile organic compound (VOC). Six alternative low-HAP or HAP-free commercial adhesives were evaluated using high-pressure decorative plastic laminate bonded to fir plywood. Testing included edge lift, open time, and shear strength in accordance with the A-A-1936A specification. No alternatives passed all of the requisite tests in the specification, although a few adhesives passed two out of the three requirements. In particular, 3M-94 CA is the most promising alternative adhesive and should only require minor modifications to meet the specification requirements.

15. SUBJECT TERMS
SPOTA, HAP-free, VOC, adhesive, open time, shear strength, edge lift, A-A-1936A

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1. Introduction

The U.S. Environmental Protection Agency (EPA) is considering proposing the Defense Land Systems and Miscellaneous Equipment (DLSME) National Emission Standard for Hazardous Air Pollutants (NESHAP) that will affect U.S. Army surface coating operations (*1*). The materials used for coating operations at many U.S. Army installations were surveyed, and it was determined that the Army used numerous adhesives and sealants, among other coating materials, that contain significant amounts of hazardous air pollutants (HAPs). The Army has determined that it is more cost-effective to reduce or eliminate HAPs from coating formulations rather than using emissions control devices to capture and treat them (*1*). Therefore, the goal of the Sustainable Painting Operations for the Total Army (SPOTA) program is to significantly reduce the amount of HAP emissions produced in coating operations, including adhesives and sealant application and removal. Adhesives and sealants account for approximately 5% of Army-wide HAP emissions from surface coating operations (*1*).

A-A-1936A is a common contact adhesive specification used for commercial and military applications. This specification describes the commercial types of neoprene adhesive suitable for contact bonding of many materials such as leather, wood, fabrics, unglazed ceramics, wall boards and carpet (*2*). Adhesives may be used to bond plastic decorative laminates to wood and metal surfaces as well. These adhesives are intended to be applied by brush or spray.

Surveys of Army installations during 2003 indicated that A-A-1936A contact adhesive produced 33% of the Army HAP emissions amounting to 1383 pounds (lbs) of HAP emissions in 2003 (*1*). In fact, this adhesive typically is composed of 15–25 weight-percent (wt.%) of toluene and hexane HAPs. However, while working on this project, we tried to determine actual uses of this adhesive in the Army. Although there were significant Contiguous United States (CONUS) applications of this adhesive in 2003, little military domestic use of this A-A-1936A adhesive could be found in 2009 (*3*). Yet, this adhesive was identified as having significant use in Iraq operations (*3*). The applications indicated for these operations include bonding table top laminates to wood platforms (*3*).

This work focused on determining a suitable HAP-free or low-HAP contact adhesive replacement for the A-A-1936A specification. We identified various commercially available, environmentally friendly adhesives with promising characteristics through a survey of products from adhesive manufacturers (*1*). This work assessed the performance of these alternatives according to the requirements of the A-A-1936A specification.

2. Materials

Two commercial contact adhesives, 3M Scotch-Weld* 10 and Clifton CC-5527, claim to meet the A-A-1936A specification and were used as baseline materials. Four HAP-free and two low-HAP contact adhesives were selected as possible alternatives for this research. Table 1 contains a summary of the physical properties for the adhesives used in this test series. We worked with various adhesive companies, including 3M and Clifton to identify the most suitable alternatives. The alternatives described below are those agreed upon by the U.S. Army Research Laboratory (ARL) and these companies.

Table 1. Summary of physical properties for adhesives in test series.

Adhesive Name	Type	Solvents	Solids (wt.%)	HAP (wt.%)	VOC (g/L)	Density (lb/gal)
3M Scotch-Weld 10 Contact Adhesive	Baseline	Petroleum Distillate, Acetone, Toluene and n- Hexane	21–25	22	422	6.9 ± 0.2
Clifton CC-5527 Contact Cement (neutral)	Baseline	Acetone, Toluene, Hexane	22 ± 2	15–25	683	6.83
3M Scotch-Weld 847	HAP free alternative	Acetone	33–39	0	0	7.4–7.8
3M Scotch-Weld 4491	HAP free alternative	Acetone, Cyclohexanone	22–26	0	43	7.0–7.4
3M Fastbond[a] 100 Foam Adhesive (neutral)	HAP free alternative	Water	45–49	< = 0.5	13	9.0–9.4
3M Scotch-Weld 94-CA Hi-Strength Adhesive (red)	HAP free alternative	Methyl Acetate	34	0	0	8.03
3M Fastbond[a] 30-NF Contact	Low HAP alternative	Water, Methanol, Toluene	47–51	4–5	37	8.9–9.3
DAP Weldwood[b] Nonflammable Contact Cement (clear)	Low HAP alternative	Water, Toluene	53–55	1–5	<80	9.3

[a] 3M Fastbond is a registered trademark of 3M.
[b] DAP Weldwood is a trademark of DAP Products Inc.

3M Scotch-Weld Neoprene Contact Adhesive 10 (baseline) is a multipurpose brushable contact adhesive, which may be used to bond plastic laminate, aluminum, steel, wallboard, wood masonry, rubber, and canvas (*4*). This adhesive contains 22 wt.% HAPs and 422 g/L volatile organic compounds (VOCs) (*5*). The HAPs are petroleum distillate, n-hexane, and toluene. Also, this product contains cyclohexane (VOC), and acetone (HAP and VOC exempt). This adhesive has a bonding range of 60 minutes (min) and meets the specification requirements of MMM-A-121, MMM-A-130B, and A-A-1936A (*4*). Note that MMM-A-130B was cancelled April 12, 1996 and superseded by A-A-1936A (*6*).

*3M Scotch-Weld is a registered trademark of 3M.

Clifton Adhesive CC-5527 (baseline) is a fast drying brush and roller grade polychloroprene rubber-based contact cement, which complies with A-A-1936 Type I (*7*). It cures at room temperature and is designed to bond decorative plastic to hardboard, wood, metal, leather, fabrics, carpet backing, and other materials. This adhesive contains approximately 16–25 wt.% HAPs (toluene and hexane) and approximately 567 g/L VOCs (*8*). Also, this product contains heptane (VOC), and acetone (HAP and VOC exempt).

3M Scotch-Weld Nitrile High Performance Rubber Gasket Adhesive 847 (alternative) is a medium viscosity grade for brush or flow application while providing strong, flexible bonds. 3M-847 provides excellent resistance to many fuels and oils (*9*). It bonds leather, nitrile rubber, most plastics, and gasketing materials to a variety of substrates (*9*). The carrier solvent is acetone 40–70 wt.% (HAP and VOC exempt) (*10*).

3M Scotch Weld Nitrile Industrial Adhesive 4491 (alternative) dries fast while providing a strong and flexible bond, is resistant to weathering, water, fuels, and oil (*11*). This product bonds foams, plastics, vinyl extrusions, and sheeting. This adhesive contains acetone (HAP and VOC exempt) 65–75 wt.% and cyclohexanone 3–7 wt.%, which is a VOC but non-HAP (*12*).

3M Fastbond Foam Adhesive 100 (alternative) is a one-part, water-dispersed, fast-setting adhesive, This neoprene-based product bonds many types of flexible polyurethane foam, latex foam fabric, polyester fiberfill, wood, plywood, plywood, particleboard, and many plastic and metal surfaces (*13*). This adhesive contains 0 wt.% HAPs and 0.11g/L VOCs, less H_2O and exempt solvents (*14*).

3M Scotch-Weld Hi-Strength Post-Forming Adhesive 94 CA (alternative) is an industrial grade general purpose product formulated for high coverage and excellent spray control (*15*). This product is 60–70 wt.% methyl acetate (HAP-free and VOC exempt), contains 0–0.7 wt.% HAPs, 0–2 g/L VOCs, less H_2O and exempt solvents and has a synthetic elastomer base (*16*).

3M Fastbond Contact Adhesive 30NF (alternative) is water dispersed and sprayable with a polychloroprene base. This product is nonflammable in the wet state and has a polychloroprene base. It bonds most foamed plastics, plastic laminate, wood, plywood, and canvas to themselves or each other (*17*). This adhesive contains 30–60 wt.% water and 2–10 wt.% methyl alcohol and toluene; both are HAPs and VOCs (*18*).

DAP Weldwood Nonflammable Contact Cement (alternative) is a high-solids polychloroprene-based, waterborne contact adhesive. It bonds decorative laminate, paper, cork, cloth-backed vinyl, drywall, wood, and plywood (*19*). This product contains toluene HAP/VOC at 1–5 wt.% (*20*).

3. Experimental Method

3.1 Edge Lift Test

Prior to adhesive application the bonding surfaces were cleaned of any dirt or debris. Two coats of adhesive, allowing drying between coats, were applied by brush to a 30 cm × 30 cm piece of 1.5-mm-thick plastic high pressure decorative laminate (HPDL) and to a 30 cm × 30 cm × 1.2 cm piece of fir plywood. The substrates were bonded together and a 2-inch (in)-wide hand roller was used to remove entrapped air and facilitate adhesion. The assemblies were aged for 24 hours (h) at room temperature. The specimens were heated in an air circulating oven for 2 h at 60 °C and allowed to cool at standard conditions. The difference in the coefficients of thermal expansion between the two substrates causes a significant shear force that can cause delamination of the adhesive bond. The edge lift test measures the ability of the adhesive to prevent delamination of assembly simply through a measurement of the delamination length. To meet the specification for A-A-1936, the distance that the HPDL substrate has separated from the plywood shall not exceed 1.5 mm (*2*).

3.2 Open Time Test

The bonding surfaces were cleaned of any dirt or debris prior to adhesive application. Two coats of adhesive, allowing drying between coats, were applied by brush to a piece of 1.2-cm-thick fir plywood and matching piece of 1.5-mm-thick HPDL. The substrates were dried for 1 h at room temperature, then bonded together so that 1 in of the laminate extended over the plywood on two sides. A 2-in-wide hand roller was used to remove entrapped air and establish adhesive bond. Immediately following the hand roller step, the HPDL substrate shall not peel from the plywood (*2*). This test measures the aggressive tack of the adhesives. Aggressive tack is the property of certain adhesives, particularly non-vulcanizing rubber adhesives, to adhere on contact to themselves at a stage in the evaporation of volatile constituents, even though they seem dry to the touch (*22*).

3.3 Shear Strength Test

Samples were prepared in accordance with American Society for Testing and Materials (ASTM) D 1002 *Standard Test Method for Apparent Shear Strength of Single-Lap-Joint Adhesively Bonded Metal Specimens by Tension Loading* (metal to metal) with different substrates as described in the A-A-1936A specification (*2*). Each lap shear assembly consisted of one piece 1 in × 4 in fir plywood and 1 in × 3 in HPDL, and bonding surfaces were cleaned to remove any dirt or debris prior to adhesive application. Two coats of adhesive, allowing drying between coats, were applied by brush to 6-mm-thick fir plywood and 1.5-mm-thick HPDL. The substrates were bonded together and cured for a minimum of seven days at room temperature. The bonded area of overlap was 1 in × 1 in and 30 assemblies per adhesive were tested. Tensile loading was

applied at a rate of 0.05 in/min for a 1 square inch bond area (*21*). Minimum shear strength of 150 pound per square inch (psi) is required for the A-A-1936A specification (*2*).

4. Results and Discussion

4.1 Edge Lift Test

Edge lift is a type of delamination, which is the separation of the substrates due to failure of the adhesive, either in the adhesive itself or at the interface between the adhesive and the adherend (*22*). The stress of the adhesive is a result of differences in coefficient of thermal expansion between the HPDL and plywood substrates. The results of the tests showed that six of the eight adhesives passed the edge lift test. A summary of the edge lift test results are listed in table 2. As expected, both controls passed this test. 3M-4491 (alternative) and 3M-100 (alternative) both failed this test with edge lift values exceeding 1.5 mm. The remaining six adhesives passed this test, but only 3M-94 (alternative) and 3M-30NF (alternative) had an edge lift value of 0 mm (figure 1). Also, figure 1 (left) contains an example of *legging*, which is the drawing of filaments or strings when adhesive bonded substrates are separated (*22*). Note that legging can occur with passing specimens, as long as the delamination length is less than 1.5 mm.

Table 2. Summary of edge lift test results.

Test Name	Specification	3M-10	CC-5527	3M-847	3M-4491	3M-100	3M-94CA	3M-30NF	DAP-NFCC
		control	control	HAP-Free	HAP-Free	HAP- Free	HAP-Free	low HAP	low HAP
		Pass	Pass	Pass	Fail	Fail	Pass	Pass	Pass
Edge Lift	0-1.5mm of lift	0.98 mm	0.68 mm	1.41 mm	3.79 mm	3.44 mm	0 mm	0 mm	0.79 mm

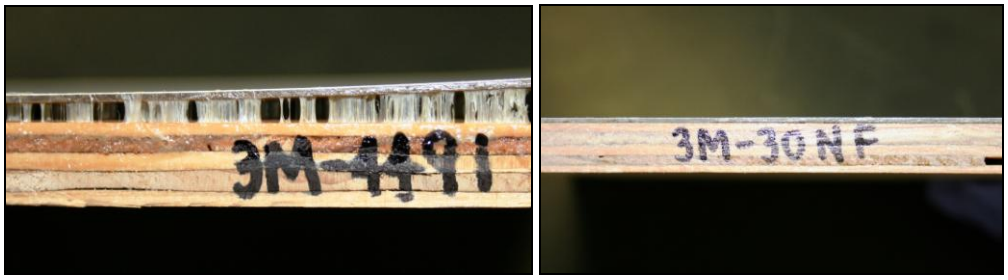

Figure 1. Edge lift failure (left) and passing (right).

4.2 Open Time Test

Open time is defined as the interval between adhesive application to the adherend and assembly of the adhesive joint (*22*). Table 3 contains a summary of the open time results for the test series. Six of the eight adhesives passed the open time test yielding failure of the HPDL substrate when trying to separate the bonded assembly. Figure 2 illustrates the HPDL substrate failure of the

assembly with 3M-10 (baseline) adhesive. 3M-100 (alternative) failed the open time test, because the substrates were easily pulled apart after bonding. Further examination of the assembly showed the failure was cohesive in nature for 3M-100 (figure 3), indicating that this adhesive takes too long to cure to meet A-A-1936A. Note results from the shear strength testing (below) indicate that weaker adhesives passed the open time test, thus indicating that cure time for 3M-100 is too long. The adhesive must remain *aggressively tacky* to form a sufficient bond. 3M-94 CA (alternative) failed the open time test because the adhesive was completely dry at the time of bonding, and the substrates did not adhere upon contact.

Table 3. Summary of open time results.

Test Name	Specification	3M-10	CC-5527	3M-847	3M-4491	3M-100	3M-94CA	3M-30NF	DAP-NFCC
		control	control	HAP-Free	HAP-Free	HAP- Free	HAP-Free	low HAP	low HAP
Open Time	adhere 1 hr after application, cannot be pulled apart	Pass-substrate failure (HPDL)	Pass-substrate failure (HPDL)	Pass-substrate failure (HPDL)	Pass-substrate failure (HPDL)	Fail-cohesive failure	Fail-did not bond	Pass-substrate failure (HPDL)	Pass-substrate failure (HPDL)

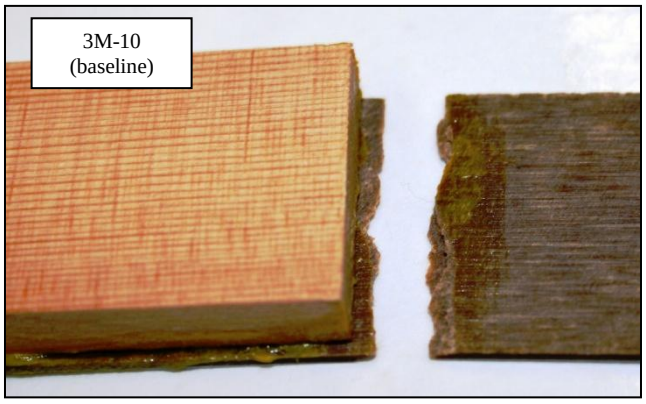

Figure 2. Open time test result (pass) with HPDL substrate failure as shown for 3M-10 (baseline) adhesive.

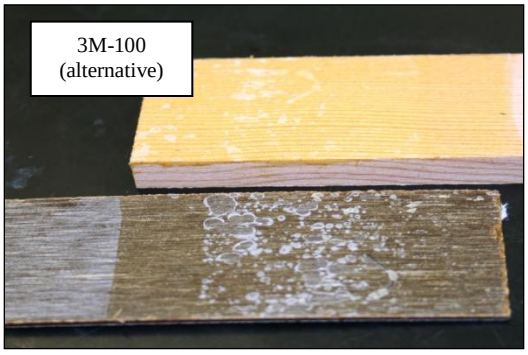

Figure 3. Open time test result (fail) with cohesive failure as shown for 3M-100 (alternative) adhesive.

4.3 Shear Strength Test

Bond strength is the unit load applied to tension, compression flexure, peel, impact, cleavage, or shear, required to break an adhesive assembly with failure occurring in or near the plane of the bond (*22*). A-A-1936A specifies the tensile shear adhesion testing method. The results of this test are summarized in figure 4, with the red dashed line indicating the minimum acceptable value according to A-A-1936A specification. 3M-10 (baseline) passed this test by a significant margin. On the other hand, the Clifton CC-5527 (baseline) adhesive did not pass, but the lap shear strength was not significantly lower than the pass criterion. Only one alternative passed this test; 3M-94 CA (alternative) had an average lap shear strength of 168 psi (figure 4). The remaining five alternative contact adhesives in the test series had shear strengths below 100 psi, and thus failed the criteria.

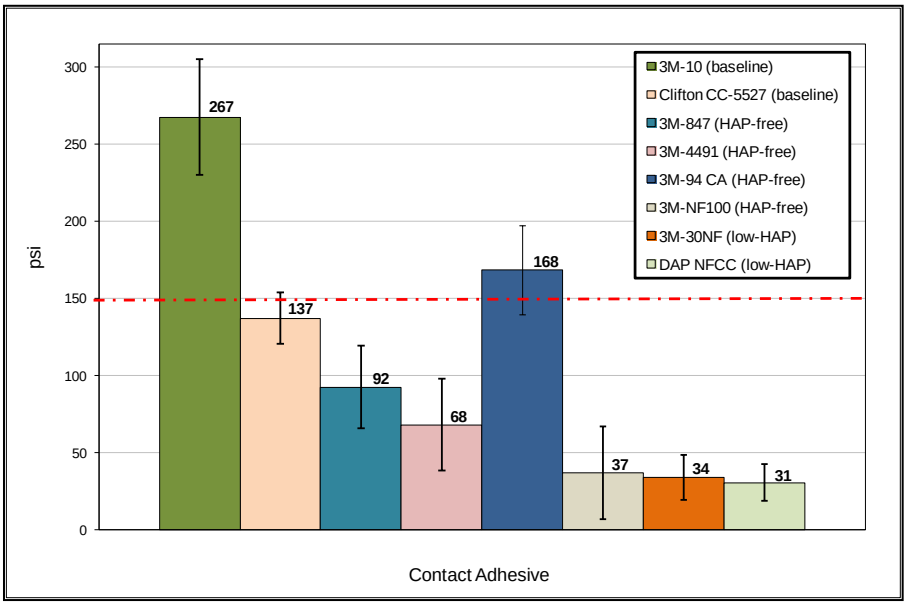

Figure 4. Average shear strength results for adhesives in test series.

4.4 Discussion

Table 4 contains a summary of all test results conducted during this research. 3M-10 (baseline) was the only adhesive to pass all three requisite tests in accordance with A-A-1936A. Clifton CC-5527 (baseline) passed two out of three tests (table 4), although it did come close to passing the lap shear test. A-A-1936A was created on April 12, 1996 and last validated on March 28, 2006. This specification does not have a qualified product list (QPL) and the baseline products were chosen because of their claim to meet the A-A-1936A requirements. Commercial formulations change frequently and if there is no required quality control process to check product performance; the performance of the product can change and no longer meet specifications.

3M-100 (alternative) was the only adhesive in this test series that failed all three test requirements. 3M-4491 was the only adhesive in this test series that failed two out of three tests. Failure of these two adhesives indicates that these adhesives are too weak to meet A-A-1936A specifications, and reformulation would need to be extensive.

The remaining four alternative adhesives failed one out of three test requirements. 3M-94 CA was the only alternative adhesive in the test series to pass the shear strength test, which is a more crucial performance specification. If open time is not critical, 3M-94 CA (alternative) could be a possible HAP-free replacement for the current A-A-1936A adhesive as it passed the other tests, unlike any of the other alternative adhesives and half of the baseline adhesives. The 3M-94 CA dried too quickly during the open time test. In our previous experience in Army bonding operations, it has been important to have significant open times because the adhesion application can take significant amounts of time due to the large parts involved (*23*). A modification to the specification could be performed relatively easily to allow reduced cure/dry times. Also, a minor formulation change could be made to the adhesive by using a solvent with a slightly slower evaporation rate—such as ethyl acetate. The second alternative to be considered would be 3M-847, as this passed the edge lift and open time and had the second highest lap shear strength of the alternatives. It is possible this lap shear strength is sufficient for actual Army applications. In which case, a modification to the A-A-1936A specification would be done to reduce the adhesion requirement. 3M-30NF and DAP NFCC had very low lap shear strengths and would likely not meet Army requirements and would require a significant reformulation effort to meet these standards.

Table 4. Summary of A-A-1936A test results.

Test Name	Specification	3M-10	CC-5527	3M-847	3M-4491	3M-100	3M-94CA	3M-30NF	DAP-NFCC
		control	control	HAP-Free	HAP-Free	HAP- Free	HAP-Free	low HAP	low HAP
Edge Lift	0-1.5mm of lift	Pass 0.98 mm	Pass 0.68 mm	Pass 1.41 mm	Fail 3.79 mm	Fail 3.44 mm	Pass 0 mm	Pass 0 mm	Pass 0.79 mm
Open Time	adhere 1 hr after application, cannot be pulled apart	Pass-substrate failure (HPDL)	Pass-substrate failure (HPDL)	Pass-substrate failure (HPDL)	Pass-substrate failure (HPDL)	Fail-cohesive failure	Fail-did not bond	Pass-substrate failure (HPDL)	Pass-substrate failure (HPDL)
Shear Strength-RT cure	150 psi (minimum)	Pass 267 psi	Fail 137 psi	Fail 92 psi	Fail 68 psi	Fail 37 psi	Pass 168 psi	Fail 34 psi	Fail 31 psi

5. Conclusions

Eight contact adhesives were included in this test series to identify a HAP-free or low-HAP replacement for federal specification A-A-1936A. No alternatives passed the requirements detailed in the specification, although a few adhesives passed two out of the three requirements. In particular, 3M-94 CA is the most promising alternative adhesive and should only require minor modifications to meet the specification requirements. Because we could not identify any significant CONUS use of A-A-1936 products in the Army, there is no strong consideration for reformulating the adhesive in our labs or through working with adhesive companies to do so or identify a more suitable alternative. For both of these reasons, we will not demonstrate/validate any of the alternative adhesives for Army applications, as the current HAP emissions from this adhesive appear to be less significant than those detailed in the 2003 report (*3*). If the use of this adhesive in the Army is found to be significant in the future, consideration will be given to reformulate these adhesives or identify more suitable alternatives. In that case, demonstration/validation on an Army platform would be necessary to ensure the alternative adhesives could meet true field/depot performance requirements.

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List of Symbols, Abbreviations and Acronyms

A-A-1936A	Federal specification for neoprene contact adhesives
ARL	U.S. Army Research Laboratory
ASTM	American Society for Testing and Materials
CONUS	Contiguous United States
DLSME	Defense Land Systems and Miscellaneous Equipment
DSI	Dynamic Sciences International, Inc.
EPA	U.S. Environmental Protection Agency
h	hour(s)
HAP	hazardous air pollutant
HPDL	high pressure decorative laminate
in	inch(es)
lbs	pounds
min	minute(s)
NESHAP	National Emission Standard for Hazardous Air Pollutants
psi	pound per square inch
QPL	qualified product list
SPOTA	Sustainable Painting Operations for the Total Army
VOC	volatile organic compound
wt.%	weight-percent

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